MEMORANDUM

TO: Mr. Addison Rice

Anderson, Mulholland and Associates

DATE: June 30, 2015

FROM: R. Infante

FILE: 1506153C

RE:

Data Validation **Air samples**

SDG: 1506153C

cleck by M 7/14/15

SUMMARY

Full validation was performed on the data for several gas samples analyzed for methane by method ASTM-D-1946 (modified): Permanent Gases and Hydrocarbons Analyzed By GC-FID. The samples were collected at the Bristol Myer Squib-Building 6 (indoor) VI facility, Humacao, PR site on June 07, 2015 and submitted to Eurofins Air Toxics, Inc. of Folson, California that analyzed and reported the results under delivery groups (SDG) 1506153C.

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: QC criteria of the ASTM method D-1946-modified. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

In general the data is valid as reported and may be used for decision making purposes. The data results are acceptable for use.

SAMPLES

The samples included in the review are listed below

Client Sample ID	Lab. Sample ID	Collected Date	Matrix	Analysis
B6-1IA-2	1506153C-01A	06/07/2015	Air	Methane
B6-2IA-2	1506153C-02A	06/07/2015	Air	Methane
B6-2IAD-2	1506153C-03A	06/07/2015	Air	Methane
B6-AA-3	1506153C-04A	06/07/2015	Air	Methane

REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- o Agreement of analysis conducted with chain of custody (COC) form
- o Holding time and sample preservation
- o Gas chromatography/mass spectrometry (GC/MS) tunes
- o Initial and continuing calibrations
- o Method blanks/trip blanks/field blank
- o Canister cleaning certification criteria
- Surrogate spike recovery
- Internal standard performance and retention times

- o Field duplicate results
- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- o Quantitation limits and sample results

DISCUSSION

Agreement of Analysis Conducted with COC Request

Sample reports corresponded to the analytical request designated on the chain-of-custody form.

Holding Times and Sample Preservation

Sample preservation was acceptable.

Samples analyzed within method recommended holding time.

GC/MS Tunes

The frequency and abundance of bromofluorobenzene (BFB) tunes were within the QC acceptance criteria. All samples were analyzed within the tuning criteria associated with the method.

Initial and Continuing Calibrations

Methane by ASTM method D-1946 (modified)

Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.

Method Blank/Trip Blank/Field Blank

Target analytes were not detected in laboratory method blanks.

Summa canister met cleaning certification criteria.

No trip/field blank analyzed with this data package.

Surrogate Spike Recovery

The surrogate recoveries were within the laboratory QC acceptance limits in all samples analyzed.

Laboratory/Field Duplicate Results

Field/laboratory duplicates were analyzed as part of this data set. Target analytes meet the RPD performance criteria of +25% for analytes $5\times SQL$.

LCS/LCSD Results

Methane

LCS/LCSD (blank spike) were analyzed by the laboratory associated with this data package. Recoveries and RPD within laboratory control limits.

Quantitation Limits and Sample Results

Dilutions were performed on ASTM-1946 (see worksheet).

Calculations were spot checked.

Certification

The following samples 1506153C-01A; 1506153C-02A; 1506153C-03A; and 1506153C-04A were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document. The results are valid. Some of the results were qualified.

Rafael Infante

Chemist License 1888



Client Sample ID: B6-1IA-2 Lab ID#: 1506153C-01A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

	Dil. Factor:	1.59	Date of Analysis: 6/15/15 03:49 PM
File Name: 9061513 Date of Collection: 6/7/15 6:04:00 PM	Dil. Factor:		Date of Collection: 6/7/15 6:04:00 PM Date of Analysis: 6/15/15 03:49 PM

 Compound
 (%)
 (%)

 Methane
 0.00016
 0.00019





Client Sample ID: B6-2IA-2 Lab ID#: 1506153C-02A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: Dil. Factor:	9061514 1.66	Date of Collection: 6/7/15 5:41:00 PM Date of Analysis: 6/15/15 04:11 PM		
Compound		Rpt. Limit (%)	Amount (%)	
Methane		0.00017	0.00020	





Client Sample ID: B6-2IAD-2 Lab ID#: 1506153C-03A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

 File Name:
 9061515
 Date of Collection: 6/7/15 5:40:00 PM

 Dil. Factor:
 1.53
 Date of Analysis: 6/15/15 04:40 PM

 Compound
 Rpt. Limit (%)
 Amount (%)

 Methane
 0.00015
 0.00019





Client Sample ID: B6-AA-3 Lab ID#: 1506153C-04A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: Dil. Factor:	9061516 1.69		ction: 6/7/15 5:49:00 PM /sis: 6/15/15 05:18 PM	
	•	Rpt. Limit	Amount	
Compound		(%)	(%)	
Methane		0.00017	0.00019	



	eurofins
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Sample Transportation Notice
Relinquishing signature on this document indicates that sample is being shipped in compliance with all applicable local, State, Federal, national, and international laws, regulations and ordinances of any kind. Air Toxics Limited assumes no liability with respect to the collection, handling or shipping of these samples. Relinquishing signature also indicates agreement to hold harmless, defend, and indemnify Air Toxics Limited against any claim, demand, or action, of any kind, related to the collection, handling, or shipping of samples. D.O.T. Hotline (800) 467-4922

180 BLUE RAVINE ROAD, SUITE B FOLSOM, CA 95630-4719 (916) 985-1000 FAX (916) 985-1020

Project Manager 18-CY aylor	·	Proie	ect Info:		Turn Around	d Lab Use	Only	X1516 (17.2)
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Lab (D) Field Sample I.D. (Location)	Can # of C	Collection	of Collection	Analyses Reques	ted Initial	Final	Receipt	Final
OF BC-1IA-2	33902 6	17/15	18:04	TO 15	-30'	1 -5.5		
02A BG-2IA-2		17/15	T	1	-30"			
03A BG-2IAD-2			17:40					andressa. Barressa
				<u> </u>	-30"			
7701 - 300 100	22509 6	17/15	17149		30"	-8.0		
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	Project Number:1506153C Date:06/07/2015
REVIEW OF VOLATILE ORGAN The following guidelines for evaluating volatile organics were actions. This document will assist the reviewer in using profe decision and in better serving the needs of the data users. The USEPA data validation guidance documents in the following "Compendium Method TO-15. Determination of Volatile Organ Specially-Prepared Canisters and Analyzed By Gas Chror January, 1999"; USEPA Hazardous Waste Support Branch. Analysis of Ambient Air in Canisters by Method TO-15, (SOP # QC criteria and data validation actions listed on the data review document, unless otherwise noted. The hardcopied (laboratory name) _EurofinsAir_Toxicsreviewed and the quality control and performance data summarize.	e created to delineate required validation assional judgment to make more informed sample results were assessed according to g order of precedence: QC criteria from hic Compounds (VOCs) In Air Collected In matography/Mass Spectrometry (GC/MS), Validating Air Samples. Volatile Organic HW-31. Revision #4. October, 2006). The worksheets are from the primary guidance data package received has been
Lab. Project/SDG No.:1506153C No. of Samples:4	Sample matrix:Air
X Holding TimesN/A_ GC/MS TuningN/A_ Internal Standard Performance	
Definition of Qualifiers: J- Estimated results U- Compound not detected R- Rejected data UJ- Estimated nondetect Reviewer: Augustus Date: 06/29/2015	···

DATA COMPLETENESS

MISSING INFORMATION	DATE LAB. CONTACTED	DATE RECEIVED
		W-V-10-1
		-
	A.	
	<u> </u>	

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		<u> </u>

All criteria were metX
Criteria were not met
and/or see below

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pН	ACTION
***************************************	**************************************			
	All samples analyzed w	vithin the recommended	method	holding time
		***************************************		NAME AND THE PARTY OF THE PARTY
		7.0100000000000000000000000000000000000		

Criteria

Aqueous samples – 14 days from sample collection for preserved samples (pH \leq 2, 4°C), no air bubbles.

Aqueous samples - 7 days from sample collection for unpreserved samples, 4°C, no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: 4 + 2 °C): N/A – summa canisters

Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimates positive results (J) and nondetects (UJ)

If the % solid of soil samples is < 10%, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted (> 10°C), estimate positive results (J) and nondetects (UJ).

DATA REVIEW WORKSHEETS

			Il criteria were metN/A ere not met see below
GC/MS TUNING			
The assessment of standard tuning QC		to determine if the sample instrumen	tation is within the
N/A_ The BFB po	erformance results wer	e reviewed and found to be within the	specified criteria.
N/A_ BFB tuning	was performed for eve	ery 24 hours of sample analysis.	
f no, use profession		mine whether the associated data sh	nould be accepted,
_ist	the	samples	affected:

If mass calibration is in error, all associated data are rejected.

All criteria were metX
Criteria were not met
and/or see below

CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:05/15/15	
Dates of continuing calibration:06/15/15	
Instrument ID numbers: GC-9	
Matrix/Level:Air/low	

DATE	LAB	FILE	CRITERIA OUT	COMPOUND	SAMPLES
	ID#		RFs, %RSD, %D, r		AFFECTED
		_	rations meet method s requirements.	pecific requirements.	Initial calibration retention
		······································	······································		

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Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC.

All %RSD must be < 15 % regardless of method requirements for CCC.

All %Ds must be < 30% regardless of method requirements for CCC.

Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of \geq 0.995 has therefore been utilized as professional judgment.

Actions

If any compound has an initial RF or a continuing RF of < 0.05, estimate positive results (J) and reject nondetects (R), regardless of method requirements.

If any compound has a %RSD > 15%, estimate positive results (J) and use professional judgment to qualify nondetects.

If any compound has a %RSD > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 30%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 30%, estimate positive results (J) and nondetects (UJ).

If any compound has a % D > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has r < 0.995, estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

All criteria were metX
Criteria were not met
and/or see below

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
All_method	 d_blank_meeth_	 _method_speci	fic_criteria	
Summa_c	anisters_met_cl	eaning_certifica	ation_criteria	
Field/Equipmen				
DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
No_field/trip/eq	uipment_blanks	_analyzed_with	n_this_data_package	

All criteria were metX
Criteria were not met
and/or see below

VB. BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is < sample quantitation limit (SQL) and \le AL, report the compound as not detected (U) at the SQL.

If the concentration is \geq SQL but \leq AL, report the compound as not detected (U) at the reported concentration.

If the concentration is \geq SQL and > AL, report the concentration unqualified.

Notes:

High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES
			-11		

All criteria were met _	_N/A
Criteria were not met	
and/or see below	

SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID

SURROGATE COMPOUND

ACTION

1,2-DICHLOROETHANEd4

Toluene- 4-BFB

d8

_Surrogate	e_standards_not_adde	ed_to_the_sample.		

QC Limits*	(Air)			
LL_to:	UL_to:			

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 80 120 % for aqueous and 70 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

All criteria were met
Criteria were not met
and/or see belowN/A

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

	PD of the compounds		o not meet the criteria. Matrix/Level:			
MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION	
	_arenot_required_as	-		-	_used_to_assess	
* QC limit	s are laboratory in-ho	ouse perfoi	mance o	criteria, LL = lowe	r limit. UL = upper lin	 nit.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

If QC limits are not available, use limits of 70 – 130 %.

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

All criteria were met
Criteria were not met
and/or see belowN/A

VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD - Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID:			Matrix/Level/Unit:			
COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION	

Actions:

^{*} If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).

^{*} If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

All criteria were metX	
Criteria were not met	
and/or see below	

VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

1. LCS Recoveries Criteria

I CS ID

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD? Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

	LCS ID	COMPOUND	% R	QC LIMIT
LCS/LCS	SD_(Blank_spike	e)_analyzed_in_this_data_i	package, recoveries a	nd RPD within
				
*				

- QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- If QC limits are not available, use limits of 70 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (i) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? Yes or No. If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

		All criteria were metX Criteria were not met and/or see below
IX.	LABORATORY DUPLICATE PRECISION	
	Sample IDs: _LCS/LCSD	Matrix:Air

Field duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD <u>+</u> 25% for air samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
	RPD v	within the me	thod performand	e criteria	
· · · · · · · · · · · · · · · · · · ·					

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

			All criteria we Criteria were and/or see be	
IX.	LABORATORY	DUPLICATE PRECISION		
	Sample IDs:	_B6-2IA-2/B6-2IAD-2	Matrix:	Air

Field duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD <u>+</u> 25% for air samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
	RPD v	vithin the met	hod performand	e criteria.	

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

All criteria were metN/A
Criteria were not met
and/or see below

X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- * Area of +40% or -40% of the IS area in the associated calibration standard.
- * Retention time (RT) within \pm 0.06 seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE ACTION RANGE	
_Internal_s _method		r_calibration;_	samples_quantitat	ed_by_the_external_standard_	
Actions:					

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%	IS AREA > + 40%
Positive results	J	J
Nondetected results	R	ACCEPT

If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

All criteria were metX
Criteria were not met
and/or see below

XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

B6-2IAD-2

Methane

RF = 157692659

[] = (19688)/(157692659)

 $= 1.25 \times 10^{-4} \%$ OK

All criteria were metX
Crîteria were not met
and/or see below

XII. QUANTITATION LIMITS

A. Dilution performed

SAMPLE ID	DILUTION FACTOR	REASONS FOR DILUTION
Dilution was pe	rformed on all samples t	by a factor of less than 2 fold.
- 1940		

B.	Percent Solids
	List samples which have ≤ 50 % solids

Actions:

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ) If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects (R)